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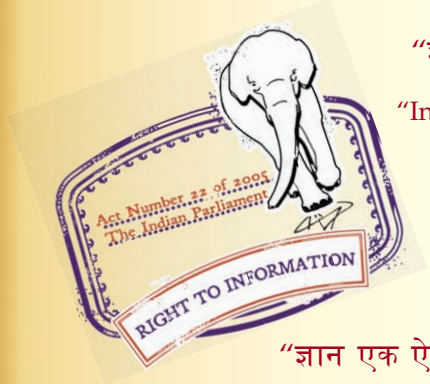

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IS : 12039 - 1986

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SPECIFICATION FOR SPENT BLEACHING EARTH

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AMENDMENT NO. 1 JANUARY 1995
TO
IS 12039 : 1986 SPECIFICATION FOR SPENT BLEACHING EARTH

[*Page* 4, *Table* 1, *Sl No.* (ii)] — Substitute '*Max*' *for* '*Min*'.

(FAD 44)

Reprography Unit, BIS, New Delhi, India

Indian Standard

SPECIFICATION FOR SPENT BLEACHING EARTH

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 28 November 1986, after the draft finalized by the Oils and Oilseeds Sectional Committee had been approved by the Chemical Division Council and the Agricultural and Food Products Division Council.

0.2 When dry triglyceride oils are bleached with clay/carbon, the spent earth cake produced from the filter press contains about 40 percent oil trapped in the bleaching earth. In order to minimize the losses of oil through spent earth, it is a practice in some industries to blow air/steam or other compressed gases to take out as much oil from the spent earth as possible. The oil removed is usually inferior to the bleached oil obtained during filtration. The removal of this oil also accelerates degradation of the remaining oil still held by the clay. Spent bleaching earth containing high levels of fatty matters, can be solvent extracted for the recovery of the oil. This is not yet very common in India. At present most of the spent bleaching earth is converted into low grade soaps, usually for utensils and dishes where the clay provides mild abrasive action. If the original oil has high iodine value, the oil present in the spent bleaching earth is likely to be oxidized and/or polymerized, thus making it less useful for soap.

0.3 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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1. SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for spent bleaching earth.

*Rules for rounding off numerical values (*revised*).

2. TERMINOLOGY

2.1 For the purpose of this standard, the definitions given in IS : 11476-1985* shall apply.

3. REQUIREMENTS

3.1 The material shall have iodine value and total fatty matter as agreed to between the purchaser and the supplier.

3.2 The spent bleaching earth shall also conform to the requirements given in Table 1 when tested according to methods prescribed in col 4, 5 6 of Table 1.

TABLE 1 REQUIREMENTS FOR SPENT BLEACHING EARTH

Sl No.	Characteristic	Requirement	Method of Test Ref to Clause No. of		Appendix A
			IS : 286-1978*	IS : 548 (Part 1)-1964†	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Moisture and volatile matter content, percent by mass, *Max*	5·0	4	—	—
ii)	Unsaponifiable matter, percent by mass on TFM, *Min*	5·0	—	8	—
iii)	Oxidised fatty acid, percent by mass on TFM, *Max*	5·0	—	—	A-2

*Methods of sampling and test for soaps (*second revision*).

†Methods of sampling and test for oils and fats : Part 1 Sampling, physical and chemical tests (*revised*).

4. PACKING AND MARKING

4.1 Packing — The material shall be packed in suitable packages/ containers as agreed to between the purchaser and the supplier.

4.2 Marking — The packages/containers shall be securely closed and marked legibly with the following information:

a) Name of the material;

b) Manufacturer's name and recognized trade-mark, if any;

c) Net mass of the material;

d) Batch number or lot number in code or otherwise; and

e) Month and year of manufacture.

*Glossary of terms relating to oils and fats.

4.2.1 The packages/containers shall also, in addition, be legibly and indelibly marked with the information required under the standards of *Weights and Measures (Package Commodities) Rules, 1977.*

4.2.2 The packages/containers may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5. SAMPLING

5.1 Representative samples of the material shall be drawn as prescribed under 3 of IS : 548 (Part 1)-1964*.

APPENDIX A

[*Table* 1, *Item* (iii)]

METHOD OF TEST FOR SPENT BLEACHING EARTH

A-1. QUALITY OF REAGENTS

A-1.1 Unless stated otherwise, pure chemicals and distilled water (*see* 1070-1977†) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF OXIDIZED FATTY ACID

A-2.0 General

A-2.0.1 A known quantity of the material is saponified with alcoholic potash and the soap formed is treated with mineral acid to release the

*Methods of sampling and test for oils and fats: Part 1 Sampling, physical and chemical tests (*revised*).

†Specification for water for general laboratory use (*second revision*).

fatty acids. The petroleum ether insoluble but ethyl ether soluble matter, which is termed as oxidized fatty acid, is determined by evaporation of the ethyl extract of the residual aqueous layer left behind after the petroleum ether extraction.

A-2.1 Apparatus

A-2.1.1 *Stoppered Glass Cylinder* — 250-ml capacity.

A-2.1.2 *Separating Funnel* — 500-ml capacity.

A-2.1.3 *Flasks* — 250-ml capacity, flat bottom; and 300-ml capacity, conical.

A-2.2 Reagents

A-2.2.1 *Alcoholic Potassium Hydroxide Solution* — Dissolve 50 g of potassium hydroxide in 1 litre of 95 percent ethyl alcohol.

A-2.2.2 *Dilute Hydrochloric Acid* — 1 : 1 by volume.

A-2.2.3 *Methyl Orange Indicator* — Dissolve 0·1 g of methyl orange indicator in 100 ml of water.

A-2.2.4 *Petroleum Ether* — 60°C/80°C distillation range.

A-2.2.5 *Ethyl Ether*

A-2.3 Procedure

A-2.3.1 Weigh accurately 3 to 5 g of the fatty matter into a 300-ml conical flask. Add 50 ml of alcoholic potash, cover with an inverted funnel and heat on a water-bath to saponify. Agitate frequently and heat for at least 30 minutes or until saponification is complete.

A-2.3.2 Remove the watch-glass and continue heating on a water-bath with occasional agitation to evaporate the alcohol. To avoid oxidation, do not evaporate beyond a pasty mass. If necessary, add a small amount of water when most of the alcohol have evaporated.

A-2.3.3 Add 100 ml of distilled water and heat until the soap has completely dissolved. Wash the contents into a glass-stoppered cylinder with hot distilled water, taking care not to exceed a total volume of 130 ml in the cylinder.

A-2.3.4 Add 3 to 5 drops of indicator and neutralize with hydrochloric acid to the pink methyl orange end point. Then add 1 ml of excess acid. Rotate the cylinder gently to mix the contents.

A-2.3.5 Cool to at least 35°C and add 125 ml of petroleum ether. The fatty acids need not have cleared completely before adding the ether. Stopper the cylinder, shake gently and allow to stand until the petroleum ether layer separates.

A-2.3.6 Siphon the petroleum ether layer into a 500-ml separating funnel, making sure that as little as possible of the insoluble matter which gathers at the ether-water interface is carried over into the separating funnel. If any appreciable amount of insoluble matter does siphon over into the separating funnel, it will usually settle to the bottom and shall be drained back into the extracting cylinder. Make at least 4 more similar extractions using 25 to 30 ml of petroleum ether, shaking the cylinder vigorously for 30 seconds with each extraction. Extractions shall be continued until the petroleum ether layer is practically colourless.

A-2.3.7 To the acid water remaining in the extraction cylinder, add 25 to 30 ml of ethyl ether, stopper, shake gently and allow to stand until the ether layer separates. Siphon the ethyl ether layer through a filter paper into a tared 250-ml flat bottom flask which has been dried and cooled in a desiccator. Make at least 4 more similar extractions using 25 to 30 ml of ethyl ether each time, and shaking the cylinder vigorously for 30 seconds with each extraction. The last ethyl ether extract shall be practically colourless.

A-2.3.8 Filter all extracts through the same filter paper and finally wash this filter paper thoroughly with ethyl ether to recover all the oxidized acids.

A-2.3.9 Evaporate the ethyl ether extracts on a water-bath under a gentle stream of clean dry air. Finally, dry the oxidized fatty acids in an air oven at 105° ± 2°C for 30 minutes. Cool in a desiccator to room temperature and weigh. Repeat until constant mass, that is, to within 0·1 percent between successive weighings, is obtained.

A-2.4 Reporting — Report the oxidized fatty acids as a percentage of the material taken for the test.

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

Quantity	*Unit*	*Symbol*
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

Quantity	*Unit*	*Symbol*
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

Quantity	*Unit*	*Symbol*	*Definition*
Force	newton	N	1 N = 1 kg,m/s^2
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m^2
Frequency	hertz	Hz	1 Hz = 1 c/s(s^{-1})
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m^2

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